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IS 6406: 1994

भारतीय मानक

चमकीला नीला एफसीएफ, खाद्य ग्रेड — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

BRILLIANT BLUE FCF, FOOD GRADE — SPECIFICATION

(Second Revision)

UDC 664·099·6: 667·283·4-126

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

This standard is one of a series of Indian Standards on synthetic food colours permitted under the *Prevention of Food Adulteration Rules*, 1955 issued by the Ministry of Health, Government of India. These rules, *inter-alia* prescribe:

'All food colours including natural colouring matter and permitted synthetic food colours and their preparations or mixtures excluding saffron and curcumin shall be sold only under the BIS Certification Mark.'

'Indian Standard specification for fast green FCF' (IS 6406:1971) was first issued in 1971. It was revised in 1977 to bring it in line with the FAO/WHO specifications after taking into account the indigenous data generated. This standard is being revised taking into consideration the latest FAO/WHO specification for the food colour issued by FAO/WHO [see FAO Food and Nutrition Paper 31/1 (1984) 'Specification for identity and purity of food additives'], latest specifications laid down under Food Chemical Codex, EEC Directives, Canadian Food Laws and Food and Drugs Act of USA. In addition, due consideration has been given to the indigenous data.

In this revision limits for various dye intermediates, chromium and heavy metals have been added to align it with International requirements.

While formulating this standard, necessary consideration has been given to the Standards of Weights and Measures (Packaged Commodities) Rules, 1977. The standard is, however, subject to restrictions imposed under these Rules wherever applicable.

Brilliant blue FCF is hygroscopic in nature and its shade changes with different pH. Suitable precautions should, therefore, be taken in packing the colour.

Colour brilliant blue FCF is described below:

Common Name - Brilliant blue FCF.

Synonyms - C.I. Food Green 2, FD and C Blue No. 1. Blue brilliant FCF.

Class -- Triarylamethane.

Colour - Blue.

Colour Index — (1975) No. 42090.

Chemical Name — Disodium salt of 4-[{ 4-(N-ethyl-p-sulfobenzyl-amino)-phenyl }-(-2-sulphonium-phenyl)-methylene]-{ 1-(N-ethyl-N-p-sulph-benzyl)- △ 2, 5-cyclohexadienimine }.

Empirical Formula - C₃₇H₃₄N₂Na₂O₉S₃.

Molecular Weight - 792.86.

Structural Formula

(Continued on third cover)

Indian Standard

BRILLIANT BLUE FCF, FOOD GRADE — SPECIFICATION

(Second Revision)

1 SCOPE		IS No.	Title
1.1 This standard prescribes the requirements and methods of test for brilliant blue FCF, food grade.		1699 : 1994	Methods of sampling and test for food colours (second revision)
2 REFERENCES		2491 : 1972	Code for hygienic conditions for
The following Indian Standards are necessary adjuncts to this standard:			food processing units (first revision)
IS No.	Title	3 REQUIREMENTS	
1070 : 1992	Reagent grade water (third revision)	3.1 The material shall conform to the requirements prescribed in Table 1.	

Table 1 Requirements for Brilliant Blue FCF

SI No.	Characteristic	Requirement	Method of Test, Ref to		
			Annex of This Standard	Clause of IS 1699 -: 1994	
(1)	. (2)	(3)	(4)	(5)	
i)	Total dye content, corrected for sample dried at $105 \pm 1^{\circ}$ C for 2 h, percent by mass, <i>Min</i>	85	Λ	_'	
ii)	Loss on drying at 135°C, and chlorides and sulphates expressed as sodium salt, percent by mass, <i>Max</i>	15	-	6 13 14	
iii)	Water-insoluble matter, percent by mass, <i>Max</i>	0.2	-	7	
iv)	Combined ether extracts percent by mass, Max	0.2	-	8	
v)	Subsidiary dyes, percent by mass, Max	3	В	_	
vi)	Dye intermediates, percent by mass, <i>Max</i>				
	a) O, sulpho-benzaldehyde, Max	1.5	C	-	
	b) N-N' ethyl-benzyl-aniline-3- sulphonic acid, <i>Max</i>	0.3	C	12	
	c) Leuco base, percent by mass, <i>Max</i>	5	D	-	
vii)	Heavy metals, (as Pb), mg/kg, <i>Max</i>	40	-	16	
	- Lead, mg/kg, Max	10	~	15	
	- Arsenic, mg/kg, Max	3	-	15	
	- Chromium, mg/kg, Max	50	_	15	

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3.2 Freedom from Contaminants

Precautions shall be taken to ensure that the material is free from aromatic amines, aromatic nitro compounds, aromatic hydrocarbons, and cyanides.

3.3 The product shall be processed, packed, stored and distributed under hygienic conditions in licensed premises (see IS 2491: 1972).

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in glass containers, metal containers, polyethylene containers or cardboard containers suitably lined with polyethylene. Subject to agreement between the purchaser and the vendor, other suitable containers may also be used.

4.2 Marking

- **4.2.1** Each container shall be legibly and indelibly marked with the following information:
 - a) The words 'FOOD COLOUR',
 - b) Common name of the colour,
 - c) Chemical name of the colour,
 - d) Colour index number,
 - e) Source of manufacture,
 - f) Date of manufacture,
 - g) Minimum mass in grams or kilograms,
 - h) Batch or code number,
 - j) Names of major dye intermediates present, and

k) Any other requirements as specified under the Standards of Weights and Measures (Packaged Commodities) Rules, 1977/Prevention of Food Adulteration Rules, 1955.

4.2.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

4.2.2.1 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material for tests shall be drawn and criteria for ascertaining conformity to the requirements of this specification shall be determined according to the method prescribed in 4 of IS 1699: 1994.

6 TESTS

6.1 Tests shall be carried out as prescribed in col 4, 5 and 6 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (see IS 1070: 1992) shall be employed in tests.

NOTE - 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

ANNEX A

[Table 1, Item (i)]

DETERMINATION OF TOTAL DYE CONTENT

A-0 GENERAL

A-0.1 Two methods, spectrophotometric method and titanium trichloride method, are specified. In case of dispute, spectrophotometric method shall be regarded as the reference method.

A-1 SPECTROPHOTOMETRIC METHOD

A-1.1 Apparatus

Suitable spectrophotometer with properly calibrated scale for both wavelength and optical density. However, suitable spectrocolorimeter properly calibrated against spectrophotometer may also be used.

A-1.2 Reagents

A-1.2.1 Ammonium Acetate Solution

200 mg of ammonium acetate in one litre of water (re-distilled).

A-1.3 Procedure

Weigh accurately about 100 mg of the dye sample and dissolve with ammonium acetate solution in a 250-ml volumetric flask. Dilute this with the same solvent to make a final concentration of 0.2 mg per 100 ml (approximately). Find out the optical density of this diluted solution against ammonium acetate solution as blank at 630 nm in a glass cell with 10.0 mm light path.

A-1.3.1 Simultaneously weigh accurately about 2 g of the dye sample and dry this in an air-oven at $105 \pm 1^{\circ}$ C for 2 hours. Calculate the loss of mass on drying; and from this data calculate the dry mass of the sample (M) in the final solution taken for measurement of the optical density.

A-1.4 Calculation

Total dye content in the sample, = $\frac{OD \times 100}{M \times 1640}$

where

OD = optical density found;

M = dry mass of the sample in 100 ml solution;

 $1640 = E^{1\%}_{1 \text{ cm}}$ 1 cm, 630 nm for brilliant blue FCF in ammonium acetate solution.

A-2 TITANIUM TRICHLORIDE METHOD

The method given in 5 of IS 1699: 1994 shall be followed. The percentage of total dye content shall be determined using the following calculations.

1 ml of 0.1 N titaneous

0.039 64 g of Brilliant Blue FCF.

ANNEX B

[Table 1, Item (v)]

DETERMINATION OF SUBSIDIARY DYES

B-1 PROCEDURE

The method given in 9 of IS 1699: 1994 using the

following absorptivities shall be used:

Developing solvent No. 4.

Develop chromatogram for approximate 20 h.

ANNEX C

[Table 1, Item (vi)]

DETERMINATION OF DYE INTERMEDIATES

C-0 GENERAL

C-0.1 Brilliant blue FCF is prepared by condensing 0-sulpho-benzaldehyde with *N-N'* ethyl-benzyl-aniline-3-sulphonic acid. After the completion of the reaction the traces of these intermediates remain, which can be estimated by ascending paper chromatography.

C-1 APPARATUS

C-1.1 Chromatography Tank and Ancillary Equipment

As given under 7.2.1 of IS 1699: 1994.

C-1.2 Microsyringe

Capable of 0.2 ml with a tolerance of \pm 0.000 2 ml.

-C-1.3 Ultraviolet Lamp — at 365.5 nm.

C-1.4 Filter Paper — Whatman No. 1 or equivalent.

C-2 REAGENTS

C-2.1 Ammonium Hydroxide Solution — sp gr 0.923.

C-2.2 O-Sulpho-Benzaldehyde

C-2.3 N-N'-Ethyl-Benzyl-Aniline-3-Sulphonic Acid

C-2.4 Developing Solvent — ammonium sulphate (20 percent m/v).

C-2.5 2 : 4 Dinitro Phenyl-Hydrazine Hydrochloride

- 0.1 percent solution in water.

C-3 PROCEDURE

C-3.1 Preparation of Solutions

C-3.1.1 Prepare 2 percent m/v solution of the dye in a mixture of nine parts of water and one part of the ammonium hydroxide.

C-3.1.2 Reference Substances

- a) Prepare on 100 percent basis, m/v solution of O-sulpho-benzaldehyde in a mixture of nine parts of water and one part of ammonium hydroxide, so as 5 μl of the solution shall correspond to 1.5 percent in the dye sample.
- b) Prepare on 100 percent basis, m/v solution of N-N'-ethyl-benzyl-aniline sulphonic acid in a mixture of nine parts of water and one part of ammonium hydroxide, so as 5 μl of the solution shall correspond to 0.3 percent in the dye sample.

C-3.2 Test Substances

C-3.2.1 Dye Solution

 $50 \mu l$ of the solution shall be equivalent to 1 000 micrograms of the sample.

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C-3.2.2 Reference Substances

- a) O-sulpho-benzaldehyde, and
- b) N-N'-ethyl-benzyl-aniline-3-sulphonic acid.
 NOTE Different concentrations of dye intermediates

may be prepared, if necessary.

C-3.3 Mark out a sheet of chromatographic paper as shown in Fig. 3 of IS 1699: 1994 and spot sufficient quantity of dye sample as uniformly as possible with the help of microsyringe. Also spot reference substances (C-3.2.2). Before mounting the sheet of paper, cut off some portion of the paper below the base line to take care of any spreading of the initial spot. Pour sufficient developing solvent into the tank to bring the surface of the solvent just below the cut portion of the chromatographic paper. Take out the filter paper after the developing solvent has sufficiently moved up. Dry

in an oven at 70 to 75°C or by hair drier.

C-3.4 Detection and Comparison of the Derivatives

C-3.4.1 O-sulpho-benzaldehyde shall give orange colouration by spraying the 2:4 dinitro-phenylhydrazin hydrochloride solution. The RF values detected shall give colouration between 0.5 to 0.7 (RF) in the 20 percent developing solvent in the dye sample. Only O-sulpho-benzaldehyde separately spotted shall show RF value between 0.65 and 0.80.

C-3.4.2 N-N'-ethyl-benzyl-aniline-3-sulphonic acid shall develop colouration with Iodine in the +0.1 to -0.1 RF values.

C-3.4.3 Compare the intensities of the sample and the reference substances visually and report the contents of dye intermediates.

ANNEX D

[Table 1, Item vi (c)]

DETERMINATION OF LEUCO BASE

D-1 PROCEDURE

Weigh accurately 120 ± 5 mg of sample and proceed as directed under 12 of 1S 1699: 1994 using the following absorptivities:

Absorptivity (a) = 0.164 mg/L/cm at approximate 630 nm Ratio = 0.970 6

(Continued from second cover)

Solubility — Soluble in water.

Sparingly soluble in ethanol.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Bureau of Indian Standards

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition.

This Indian Standard has been developed from Doc No. FAD 8 (262).

Amendments Issued Since Publication

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